

**Notice of Allowability**

Application No.

10/053,646

Examiner

Rip A. Lee

Applicant(s)

THORNTON ET AL.

Art Unit

1713

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☒ This communication is responsive to December 28, 2004.
2. ☒ The allowed claim(s) is/are 21, 22, 26-28, 30, 37, 38, 40-46.
3. ☐ The drawings filed on \_\_\_\_\_ are accepted by the Examiner.
4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
  - a) ☐ All    b) ☐ Some\*    c) ☐ None    of the:
  1. ☐ Certified copies of the priority documents have been received.
  2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
  3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

\* Certified copies not received: \_\_\_\_\_.

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.  
**THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.**

5. ☐ A SUBSTITUTE OATH OR DECLARATION must be submitted. Note the attached EXAMINER'S AMENDMENT or NOTICE OF INFORMAL PATENT APPLICATION (PTO-152) which gives reason(s) why the oath or declaration is deficient.
  6. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
    - (a) ☐ including changes required by the Notice of Draftsperson's Patent Drawing Review (PTO-948) attached
      - 1) ☐ hereto or 2) ☐ to Paper No./Mail Date \_\_\_\_\_.
    - (b) ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date \_\_\_\_\_.
- Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).
7. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

**Attachment(s)**

1. ☐ Notice of References Cited (PTO-892)
2. ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
3. ☐ Information Disclosure Statements (PTO-1449 or PTO/SB/08), Paper No./Mail Date \_\_\_\_\_
4. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material
5. ☐ Notice of Informal Patent Application (PTO-152)
6. ☐ Interview Summary (PTO-413), Paper No./Mail Date \_\_\_\_\_
7. ☒ Examiner's Amendment/Comment
8. ☒ Examiner's Statement of Reasons for Allowance
9. ☐ Other \_\_\_\_\_

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### DETAILED ACTION

This office action follows a request for continued examination (RCE) under 37 § C.F.R. 1.114, filed on December 28, 2004. Applicants present amended claims 21, 37, and 40. Claims 23, 29, 31-36, and 39 were canceled, and new claim 46 was added. Claims 21, 22, 24-28, 30, 37, 38, 40-46 remain for prosecution.

### EXAMINER'S AMENDMENT

An examiner's amendment to the record appears below. Should the changes and/or additions be unacceptable to applicant, an amendment may be filed as provided by 37 CFR 1.312. To ensure consideration of such an amendment, it MUST be submitted no later than the payment of the issue fee.

The application has been amended as follows:

Cancel claims 24 and 25.

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*Examiner's note:* Independent claim 21 is drawn to a method in which the solid adsorbent is silica gel. Therefore, claim 24 and 25 (which recite use of synthetic, polyacrylic/polystyrene resin) can not depend from or limit further claim 21. Note that the subject matter of claims 24 and 25 was rejected under 35 U.S.C. 102(e) in the previous office action.

***Allowable Subject Matter***

The following is an examiner's statement of reasons for allowance: Claims 21, 22, 26-28, 30, 37, 38, and 40-46 are allowed over the closest prior art cited below.

The present invention is drawn to a method comprising selectively separating stable, hydrophobic nitroxyl radicals from a reaction mixture by hydrophobic interaction by selective adsorption onto a solid adsorbent. In one embodiment, the adsorbent is silica gel. In another aspect of the invention, the hydrophobic interaction takes place in a precipitation step wherein a  $\beta$ -cyclodextrin selectively forms a complex with the stable nitroxyl radical. In another embodiment of the invention, the hydrophobic interaction takes place in a liquid-liquid extraction, and the method further comprises adding organic solvent to the reaction mixture and transferring the nitroxyl radicals into the organic solvent. Here, the organic solvent is a C<sub>6</sub> or higher alcohol.

U.S. 6,335,464 (Ochi *et al.*) discloses a process in which TEMPO is separated adsorptively from aqueous reaction mixture by addition of a synthetic resin such as Amberlite resin XAD-2. These resins may be used as a stationary phase for chromatographic purposes. The amine oxide may be desorbed and recovered from the resin using water-miscible solvents such as THF, acetone, or lower alcohols. The reference does not teach use of  $\beta$ -cyclodextrins or silica gel as the adsorbent.

Ciriminna *et al.* (*Chem. Commun.* 2000) discloses a sol-gel entrapped TEMPO for selective oxidation of sugars. In this case, the sol-gel silica is organically modified by covalently attaching a TEMPO moiety thereto. Clearly, this is not an adsorptive phenomenon.

WO 99/47258 (Pagliaro *et al.*) discloses a sol-gel glass which has been physically doped with TEMPO. Although this is an adsorptive phenomenon, the reference does not teach use of silica gel or  $\beta$ -cyclodextrins as the adsorbent.

WO 96/36621 (Heeres *et al.*) discusses prior art which relates to immobilization of TEMPO on solid supports, but the process is not adsorptive in nature. The reference also describes a process in which TEMPO is recovered from a liquid reaction medium by simple liquid-liquid extraction with diethyl ether. The reference does not teach use of a C<sub>6</sub> or higher alcohol for extraction. Applicants have shown that use of such a solvent is effective because efficient recovery is not limited by the vapor pressure (volatility) of the nitroxyl radical.

WO 95/07303 (Besemer *et al.*) describes a process in which TEMPO is recovered from a liquid reaction medium by simple liquid-liquid extraction with diethyl ether. The reference does not teach use of a C<sub>6</sub> or higher alcohol for extraction.

Kotake *et al.* (*J. Am. Chem. Soc.* 1989) teaches use of ESR and ENDOR spectroscopy for detection of formation of inclusion complexes of nitroxyl radicals and  $\beta$ -cyclodextrin. The reference does not teach the subject matter of the instant claims in that a precipitate is not formed as a result of the hydrophobic interaction. To a lesser extent, there is no suggestion of use of  $\beta$ -cyclodextrin for recovery of nitroxyl radical from a reaction mixture.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

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***Information Disclosure Statement***

Applicants have submitted an information disclosure statement identical to that filed on January 24, 2002. Applicants request consideration of U.S. 5,817,806 (Rossi *et al.*). The reference was not considered previously. It is the examiner's position that the reference is not germane to the subject matter of the instant invention. Whereas the patent teaches use of TEMPO for oxidation reactions, there is nothing relating to recovery of the oxidant in the text. It is noted that there are literally hundreds of reference which teach use of TEMPO as an oxidant.

As such, Applicants IDS of December 28, 2004 has not been initialized since a copy exists in the record.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Rip A. Lee whose telephone number is (571)272-1104. The examiner can be reached on Monday through Friday from 9:00 AM - 5:00 PM. If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, David Wu, can be reached at (571)272-1114. The fax phone number for the organization where this application or proceeding is assigned is (703)872-9306.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <<http://pair-direct.uspto.gov>>. Should you have questions on the access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll free).



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March 30, 2005



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